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OXIDIETION STUDIES OF UNFILLED STYRENE-BUTADIENE RUBBER USED IN TANK TRACK PADS BY FT-IR AND DSC

MAMES M. SLOAN and MARIANNE T. BACHAND CLYMER RESEARCH BRANCH

November 1988

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ABSTRACT

The thermo-oxidation of unfilled styrene-butadiene rubber (SBR) was examined by Fourier transform infrared spectroscopy and differential scanning calorimetry (DSC). The process was found to follow an auto-oxidative reaction sequence, where formation of alkyl and peroxy radicals is the initial step. It was also determined that only the butadiene portion of the copolymer was oxidized, no oxidation of the styrene portion could be detected. The DSC results yielded enthalpies in the range of 90 to 180 J/g with an activation energy of 35.0 kcal/mole.



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INTRODUCTION

Industrial elastomers degrade more rapidly on continued exposure to sunlight and elevated temperatures in the presence of oxygen. ^{1,2,3} As a consequence of this oxidative degradation, progressive deterioration of physical and mechanical properties are realized.

Styrene-butadiene rubber (SBR) is presently the major component elastomer used to make track pads for the Army's M-1 Abram's tank. Replacement costs for these track pads currently run the Army over \$150 M a year. Development of high performance elastomers which can handle the operational requirements for track pads is a difficult problem. One approach is to improve the materials currently in use. The first step is to evaluate various failure modes and degradation processes occurring in the elastomers.

Elastomers with an unsaturated backbone are known to be more susceptible toward oxidative degradation. In fact, oxidative degradation is the primary method for deterioration of polymers under normal use. In this report, the thermo-oxidation of an unfilled SBR is studied. Fourier transform infrared spectroscopy (FT-IR) is used to determine the mechanism of oxidation of SBR, while differential scanning calorimetry (DSC) is used to determine the thermodynamic parameters and kinetics of oxidation.

BACKGROUND

The free radical mechanism of oxidation of elastomers proceeds through a chain reaction according to a well-established sequence of reactions.^{4,5} The scheme is as follows:

initiation:	RH R'
propagation:	$R' + O_2 \longrightarrow RO_2'$
	RO_2 + RH \longrightarrow ROOH + R
termination:	2RO ₂ — nonradical products

where RH represents a hydrocarbon, R' an alkyl radical, and RO2' a peroxy radical.

This relatively simple sequence of reactions can be complicated when oxidizable impurities are present. Also, there is a possibility that secondary processes can occur, where peroxides and free radicals undergo chain scission reactions. It is, therefore, assumed that the degree of deterioration of physical properties is generally proportional to the degree of oxidation.⁶

DATA ANALYSIS

The oxidation reaction of the SBR exhibits an exothermic maximum in the DSC thermogram. The reaction enthalpy can then be determined by calculating the area under the curve. From these curves, the thermodynamic parameters and kinetics can be determined.

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The Borchardt-Daniels⁷ method was used to determine the energy of activation. The reaction is assumed to be first order in SBR and can be expressed as follows:

$$k = Ae^{-E}a^{/RT}$$

where k is the scanning rate constant, A is a pre-exponential factor, E_a is the energy of activation for the oxidation reaction, T is the temperature maxima, and R is the general constant in the gaseous state.

This equation can be reduced to a more usable form to yield:

$$\ln k = -\frac{E_a}{R} \frac{1}{T} + \ln A$$

$$\log k = E_a \frac{2.3}{R} \frac{1}{T} + \log A.$$

This equation produces a linear graph with the slope equal to E_a(2.3)/R, from which the energy of activation can be calculated.

EXPERIMENTAL

SBR-1502 was used as the base rubber. This was obtained from the Goodyear Tire and Rubber Company. No further purification was done. The rubber was dissolved in toluene and cast as a thin film onto a KBr salt plate. The toluene was then evaporated by streaming dry nitrogen over the salt plate. The KBr plate was then incubated at specified time intervals in a thermostatted oven with an atmospheric air purge. The temperature used for this study was 120°C.

The infrared spectra were obtained on a Perkin-Elmer Model 1550 Fourier transform infrared spectrometer equipped with a Model 7500 PE computer. The spectra were transferred to a floppy disk for data reduction at a later time. The spectra were taken at 4 cm⁻¹ resolution for 16 co-added scans.

DSC measurements were taken on a DuPont Model 910 differential scanning calorimeter cell with a DuPont Model 1090 controller. The sample was weighed and placed in an aluminum pan. A purge of dry air or dry nitrogen was used depending on the environment desired. The DSC curves were produced by heating the sample from room temperature to 400°C at a heating rate of 2,5,10, and 20 degrees per minute.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy

Figure 1 shows the infrared spectrum of the SBR at time zero and three spectra at various stages of thermo-oxidation. The initial spectrum shows no indications of premature oxidation although a band at 1700 cm⁻¹ is visible. This is presumed to be an absorption due to carboxylated end groups. During the polymerization process, the end groups are capped with carbonyl groups to obtain the desired molecular weight.

The top three spectra begin to show various stages of oxidation. Strong absorptions appear at 3450 cm⁻¹, 1720 cm⁻¹, and in the 1000-1200 cm⁻¹ range. These bands are increasing with reaction time and are due to oxidation products being formed.

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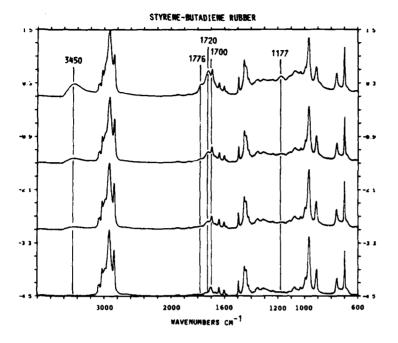


Figure 1. Styrene-butadiene rubber oxidation: progressive stages of oxidation are seen. Bottom spectrum, time zero spectrum, followed by spectra at 50 minutes, 60 minutes and 90 minutes, respectively.

Figure 2 shows two subtraction spectra of successive IR oxidation spectra. The bottom spectrum is that of 60 minutes minus 50 minutes. The top spectrum is 90 minutes minus 60 minutes. These subtraction spectra show IR band changes not seen in Figure 1. Negative bands can be observed at 3007 cm⁻¹ and 3065 cm⁻¹. Also, negative bands appear at 910 cm⁻¹ and 964 cm⁻¹. These bands are related in that they are due to the cis C-H on the double bond. The 3007 cm⁻¹ and 3065 cm⁻¹ have been assigned to the symmetric and asymmetric stretch of the = C-H. Similarly, the 910 cm⁻¹ bands are due to the vinyl C = C-H, and cis C = C-H wags, respectively. This is the first step in the oxidative process, the abstraction of the α -methylenic hydrogen. This then becomes the site of the alkyl radical.

New absorbing species become apparent at this stage. IR bands at 1050 cm⁻¹ due to C-O, at 1720 cm⁻¹ due to C=O, and at 3450 cm⁻¹ due to O-H appear. All show the incorporation of oxygen into the elastomer. The broadness of these bands demonstrate the fact that many different types of absorbing species are present. The appearance of the 1050 cm⁻¹ band is attributed to C-O absorption and most likely due to the original formation of peroxy radical groups, RO₂. The subsequent formation of ROOH is demonstrated by the appearance of the O-H species at 3450 cm⁻¹, and by the C=O group at 1720 cm⁻¹.

Figure 3 shows the major spectral changes with time. The bands at 3007 cm⁻¹, 3065 cm⁻¹ and 964 cm⁻¹ all behave in the same manner. This is clear in that they have the same shape on the graph. These are all due to the disappearance of the cis = C-H on the double bonded carbon. Similarly, the bands at 1720 cm⁻¹, 3450 cm⁻¹ and 1050 cm⁻¹ appear to be related. These are all due to various functional groups associated with incorporation of oxygen into the elastomer. The appearance of these bands is consistent with the auto-oxidation sequence found in the Background section of this report.

It is reasonable to suggest that the α -methylenic hydrogen is the hydrogen that gets abstracted. Energetically, it is the one that is most favorable. This forms the alkyl radical depicted in the auto-oxidative sequence. It becomes possible for this radical to then form a resonance structure with the adjacent double bond before attack by atmospheric O_2 . It is believed that the resonance structure can be expressed as follows:

$$C=C-C \longleftrightarrow C=C-C \longleftrightarrow C-C=C$$
.

This is the reason the IR spectra show a decrease in the 3007 cm⁻¹ and 3065 cm⁻¹ bands, those assigned to the methylenic hydrogens.

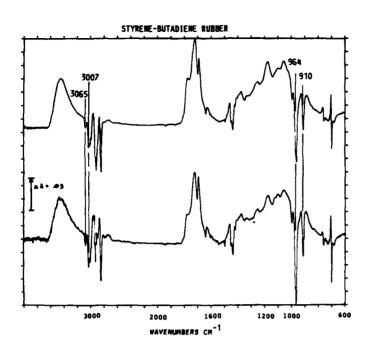


Figure 2. Difference spectra from successive oxidation spectra. Bottom, 60 minutes minus 50 minutes; Top, 90 minutes minus 60 minutes.

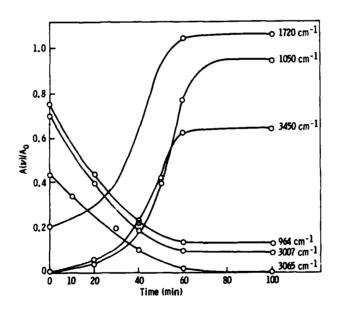


Figure 3. IR band changes with time in the oxidation of SBR at 120°C.

After the major spectral changes have manifested themselves, several small shoulders begin to appear at 1700 cm⁻¹ and 1727 cm⁻¹. These have been assigned to saturated and unsaturated aldehydes, respectively. Also, the appearance of shoulders at 1776 cm⁻¹ and 1177 cm⁻¹ are due to lactone structures caused by reorganization of the polymer backbone

and pendant side chain oxidation products. It should be pointed out that only the polybutadiene portion of the elastomer undergoes thermo-oxidation. The absence of an IR band at 3550 cm⁻¹, where ArO-H stretch would appear, demonstrates the fact that the polystyrene portion of the copolymer does not undergo thermo-oxidation.

Table 1 summarizes the IR band changes for the oxidation process.

Table 1. INFRARED BAND ASSIGNMENTS

Band	Assignment	Change	
910	Terminal C = C	-	
960	cis C=C	-	
1050	C-O	+	
1140	C-O	+	
1177	C-O, Possible Lactone Groups	+	
1700	Unsaturated Aldehyde	+	
1720	C=O, Ketonic	+	
1727	Saturated Aldehyde	+	
1776	C = O, Lactone Groups	+	
3007	cis C-H		
3068	cis C-H	-	
3450	O-H	+	

Differential Scanning Calorimetry

Figure 4 shows the DSC thermogram for SBR with a nitrogen purge. No exothermic or endothermic transition can be detected. However, if one measures the thermogram with an air purge, a clear exothermic transition can be observed between 210°C and 245°C. Figures 5 through 8 demonstrate the effect of scanning rate on enthalpy and transition temperature maximum. As one increases the scanning rate, T_{max} increases while ΔH decreases. One can then employ the Borchardt-Daniels⁷ method of determining the energy of activation. Figure 9 shows a plot of log rate versus 1/T which is linear. The slope of this line gives an activation energy of 35.0 kcal/mole. This value is somewhat larger than values previously reported. It is, however, suspected that since such a small sample size is used for the DSC measurement (10-20 mg) that the thermo-oxidation reaction is more complete. The limitations of O₂ diffusion throughout the sample is not a factor as it would be in a bulk sample.

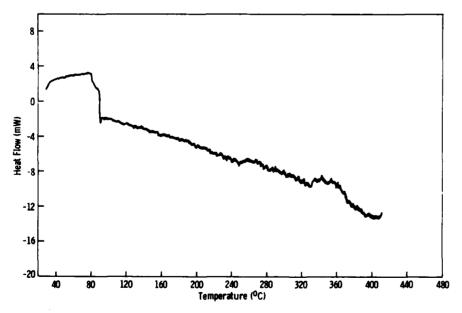


Figure 4. DSC thermogram of SBR with nitrogen purge.

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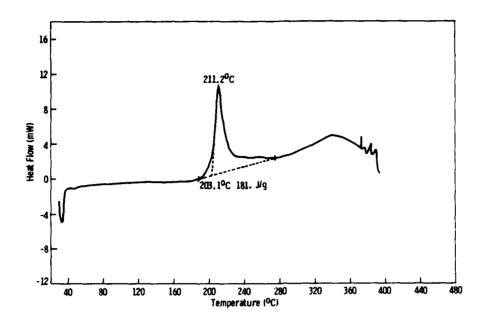


Figure 5. DSC thermogram of SBR oxidation with a scanning rate of 2°C/minute.

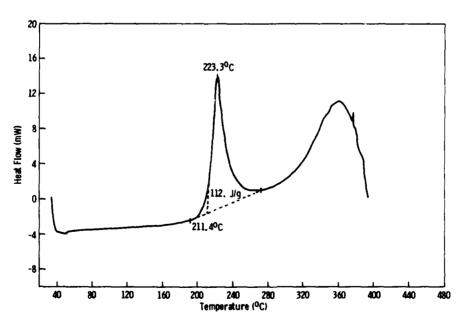


Figure 6. DSC thermogram of SBR oxidation with a scanning rate of 5°C/minute.

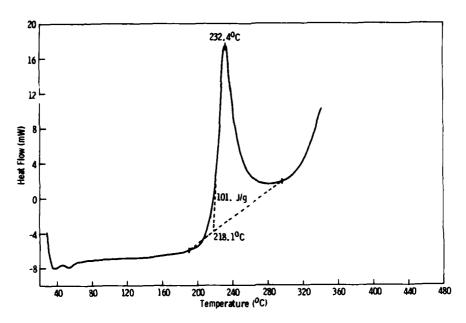


Figure 7. DSC thermogram of SBR oxidation with a scanning rate of 10°C/minute.

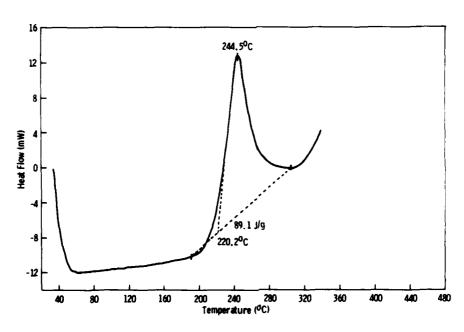


Figure 8. DSC thermogram of SBR exidation with a scanning rate of 20°C/minute.

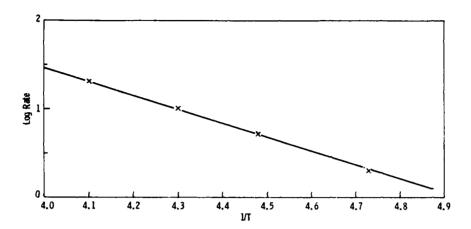


Figure 9. Borshardt and Daniels plot of iog rate versus 1/T.

CONCLUSIONS

The thermo-oxidation of styrene-butadiene rubber was studied by FT-IR and DSC. The FT-IR results suggest that the thermo-oxidation degradation is related to the auto-oxidative degradation process postulated by Cuneen⁵. The DSC results yield enthalpy values in the range 90 to 180 J/g depending on scanning rate with an energy of activation of 35.0 kcal/mole.

It was also found that the degradation is localized only to the polybutadiene portion of the copolymer. No oxidation of the polystyrene was observed.

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The thermo-oxidation of unfilled styrene-butadiene rubber (SBF) was examined by Fourier transform infrared spectroscopy and differential scanning calorimetry (DSC). The process was found to follow an auto-oxidative reaction sequence, where formation of alkyl and peroxy radicals is the initial step. It was also determined that only the butadiene portion of the co-polymer was oxidized, no oxidation of the styrene portion could be detected. The DSC results yielded enthalples in the range of 90 to 180 J/g with an activation energy of 35.0 kcal/mole.

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